

## COATINGS

UDC 678.86

### EFFECT OF CERAMIC COATINGS ON THE SOUND ABSORPTION OF HEAT-SHIELD MATERIALS

S. S. Solntsev,<sup>1</sup> V. A. Rozenenkova,<sup>1</sup> and N. A. Mironova<sup>1</sup>

Translated from *Steklo i Keramika*, No. 6, pp. 21 – 24, June, 2012.

Heat-and-sound absorbing materials and a technology for fabrication, deposition and formation of ceramic coatings, which makes it possible to obtain glass ceramic heat-and-sound absorbing materials with coatings whose properties can be regulated, have been developed. It is shown that ceramic coatings can be effectively used for heat-shield materials.

**Key words:** ceramic coatings, heat-and-sound absorbing materials (HSAM), synthesis, binder, polymers, glass fiber.

The most effective means for reducing noise in propulsion systems are multilayer, composite, noise-absorbing structures with nonmetallic honeycomb microporous fillers, which have enhanced acoustic properties and perform reliably for a long time. Combined, inorganic, microporous, blown materials are being studied as sound-absorbing layers. Two advantages of microporous materials are low mass and property uniformity over the entire surface [1 – 3].

In order to ensure the stability of the microporous structure of heat-and-absorbing materials (HSAM) and increase the temperature stability and thermal resistance it is best to use thin, flexible, sealing layers of ceramic coatings [4 – 6].

A distinguishing feature of ceramic coatings is that a protective layer of silicon-ceramic polymers is obtained. This layer is formed as a result of an organic-to-inorganic transition of a polymer on the surface of a glass fiber over the entire volume of the mat.

Ceramic coatings were synthesized on the basis of silicon-organic compositions modified with fine powders of  $\text{SiO}_2$ ,  $\text{SiC}$  and  $\text{Si}_3\text{N}_4$ . The coatings were obtained by pyrolytic breakdown of elemental-organic polymers such as polysilazanes, polycarbosilanes (PCS) and polyboro-ethoxysiloxanes (PBES) with additions of thermodynamically stable refractory oxygen and oxygen-free compounds with complex composition.

To determine recipes for ceramic-forming polymers ensuring the serviceability of coated TSAM in the temperature

range 750 – 1500°C the properties of two types of binder were studied: based on PCS with enhanced content of the silazane part and based on elemental siloxanes. Thermogravimetric analysis (DTGA) established that increasing the polysilazane content of the binder increases the yield of cross-linked polymer.

Chemical analysis of polycarbosilane binders performed by pulsed heating to 2000°C in an inert gas followed by chromatographic analysis of the products formed shows that the product of pyrolysis corresponds to the following composition:  $2\text{SiC}$ ,  $0.2\text{SiO}_2$  and  $0.2\text{Si}_3\text{N}_4$  (in moles).

The PBES binder was obtained on the basis of the synthesized oligomer. The physical-chemical characteristics of the binder are as follows: viscosity determined with a VZ-246 viscosimeter (2 mm nozzle) 40 – 60 sec, mass fraction of nonvolatile substances 55 – 60%, Si content 18.5 – 20%, B — 1.1 – 1.15% and OH-group 1.5 – 1.6%. The yield of insoluble polymer at 200°C was higher than 80%. The thermal oxidative breakdown of PBES polymer was studied in two regimes: dynamic to 1000°C and static at 750°C. The mass losses in the dynamic regime do not exceed 10%, while the losses under isothermal conditions reach 20%, most of the losses occurring during the first 5 h of heating.

The following characteristics were determined in the present work: the quality of the initial materials, method used to prepare the materials, coating deposition and formation, temperature stability of coated TSAM, phase stability, mechanical properties, and sound absorption coefficient  $\alpha$ .

The following were investigated for use as the initial components: colloidal solution of  $\text{SiO}_2$  (tetraethoxysilane

<sup>1</sup> Federal State Unitary Enterprise – All-Russia Institute of Aviation Materials (FGUP VIAM), Moscow, Russia.

**TABLE 1.** Temperature – Time Regimes for Coating Formation

Coating	Formation regime		
	Temperature, °C	Time, h	Medium
No. 16	20 – 300	5 – 8	Vacuum
	20 – 700	8 – 10	Vacuum
No. 11	20	24	Air
	200	5 – 10	Air
No. 8	20	36 – 42	Air
	80	5 – 10	Air

TEOS) and elemental-organic polymers of the type PCS and PBES, which form during pyrolytic breakdown of thermodynamically stable refractory compounds  $\text{SiO}_2$ ,  $\text{SiC}$  and  $\text{Si}_3\text{N}_4$  in air and in an inert medium. These components can form thin, protective, glass ceramic layers on the surface of glass fibers. Materials of the type ATM (siliceous, quartz, kaolinic glass fiber) were studied for use as glass-fiber substrates. These fibers are temperature-stable, inert and ecologically safe and find application as heat-and-sound absorbing materials.

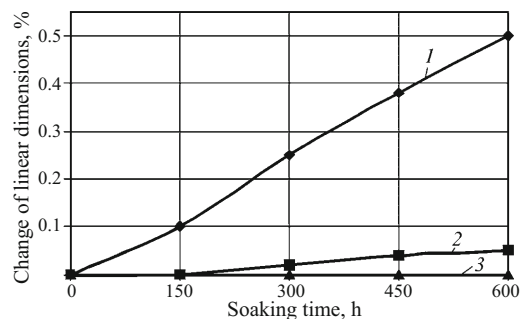
Ultrathin  $\text{SiO}_2$  fiber in the form of fabric and molded mat was used to obtain heat-and-sound insulating material. The samples possessed low density ( $0.12 - 0.8 \text{ g/cm}^3$ ) and low thermal conductivity ( $0.06 \text{ W/(m} \cdot \text{K)}$  at  $20^\circ\text{C}$ ,  $0.25 \text{ W/(m} \cdot \text{K)}$  at  $1200^\circ\text{C}$ ), were ecologically safe and resisted chemical and biological attack, which is very important for aerospace engineering.

The technological parameters for the deposition of a ceramic coating on glass fiber substrates (ATM type) with different density and structure were studied.

Since low density is one of the factors determining the sound absorption of a material, working solutions with low viscosity and low concentration of the solid phase  $\text{SiO}_2$  and  $\text{SiC}$  were studied in the present work. The following variants of coating deposition were studied: free and vacuum permeation over time from 2 to 8 h. The criterion for evaluating the coating deposition quality was the density of the samples obtained. To attain the minimum density of the samples to  $160 \text{ kg/m}^3$  the free-permeation time was 3 – 5 h; for vacuum deposition the coating deposition time was decreased by 30 – 50% to 1 – 3 h. The minimum sample density was obtained with  $140 - 160 \text{ kg/m}^3$  coatings and one-time permeation with the working solutions with polymer mass content to 5% irrespective of the coating deposition method.

The temperature – time regime of coating formation was studied. To prevent crystallization and decrease lineal shrinkage of glass fiber substrates during molding two temperature – time regimes were studied. For coating formation in such regimes the shrinkage of glass fiber substrates was less than 0.1%.

X-ray structural studies were performed on samples of ceramic coatings with and without modifiers being added.

**Fig. 1.** Shrinkage of HSAM samples versus the soaking time at  $750^\circ\text{C}$ : 1) no coating; 2) with coating No. 16, one layer; 3) with coating No. 16, three layers.

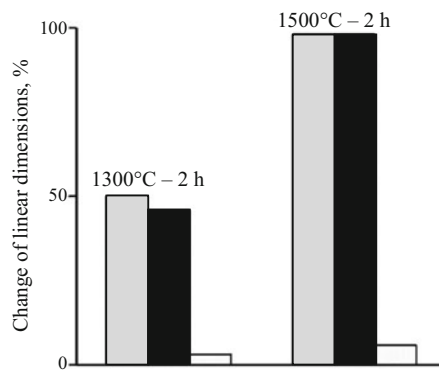
Diffraction patterns were obtained with a D/MAX-2500 diffractometer (Rigaki, Japan) in a working regime with 40 kV and 300 mA and scanning range  $2\theta = 5 - 90^\circ$ . The results of x-ray phase analysis of the coatings attests to the presence of the amorphous phases of  $\text{SiO}_2$ ,  $\text{SiC}$ ,  $\text{Si}_3\text{N}_4$ , and  $\text{SiOB}$  and the absence of cristobalite. The data obtained show that no phase transformations leading to softening of the coating and material occur during the coating formation process.

Scanning electron microscopy was used to study the microstructure of samples with a ceramic coating. The studies were performed with a JSM-840 SEM in the secondary electron regime with magnification from  $\times 200$  to  $\times 5000$ . The results showed that the ceramic layer comprises a nanostructural graded coating with a scaly structure.

The temperature stability of the samples of heat-and-sound absorbing material with a ceramic coating were studied at  $1300^\circ\text{C}$  with soaking time 2 h,  $1500^\circ\text{C}$ , 2 h and  $750^\circ\text{C}$ , 500 h (the tests were performed in a laboratory furnace with periodic weighing every 5, 10 and 25 h). The criterion for evaluating the temperature stability of the samples was the change in the linear dimensions and the surface quality of the samples. The test results are presented in Figs. 1 and 2.

The studies showed that the HSAM sample with coating No. 16 remains temperature-stable to  $750^\circ\text{C}$  for 600 h and longer. The linear dimensions of samples with a three-layer coating do not change, and no defects were found on the surface of the samples.

The shrinkage of the linear dimensions of the HSAM samples at  $1300$  and  $1500^\circ\text{C}$  with soaking to 2 h was 1 and 3% with coating No. 16, respectively, and 30 and 100% without a coating. The shrinkage of the linear dimensions of the HSAM samples with coating No. 11 at  $1300$  and  $1500^\circ\text{C}$  and soaking to 2 h was 48 and 60%, respectively. The experimental data show that the composition of the coating has a strong effect on the temperature stability of HSAM. These studies showed that the optimal composition is that of coating No. 16: it increases the temperature stability of HSAM by  $250 - 300^\circ\text{C}$ .



**Fig. 2.** Temperature stability of samples with ceramic coatings: □) HSAM without coating; ■) HSAM with coating No. 11; □) HSAM with coating No. 16.

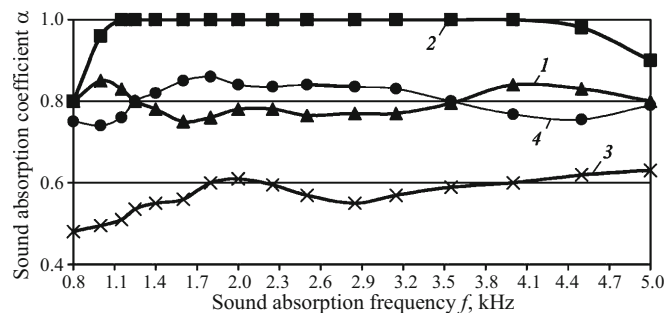
The studies of the effect of the coating composition on the mechanical properties of the HSAM samples established that the mechanical strength of the samples under compression as well as bending depends on the composition of the coating. Thus, the bending strength of the HSAM samples with coating No. 16 ( $\sigma_b = 40 - 60$  MPa) is 2 – 3 times higher than with coating No. 11 ( $\sigma_b = 20 - 30$  MPa). The compressive strength of the HSAM samples with coating No. 16 ( $\sigma_c = 10 - 21$  MPa) is 5 – 6 times higher than that of a HSAM sample with coating No. 11 ( $\sigma_c = 2 - 3.5$  MPa) and uncoated HSAM samples. Data analysis shows that the coating based on polycarbosilanes enhances the mechanical properties of the experimental samples in the initial state and after the samples were tested for temperature stability at temperatures 750 – 1500°C. A coating based on polycarbosiloxanes enhances the mechanicals of heat-and-sound absorbing materials in the initial state and after temperature-stability tests only to 750°C.

Samples of HSAM with a ceramic coating with combined perforation different with respect to form and occupied volume in the material were studied. The experiments were performed with HSAM samples with opening porosity ranging in diameter from 1.5 to 4 mm and perforation volume to 15%. The results of density measurements on the samples as a function of the perforation area are presented in Table 2.

The effects of the coating composition and perforation of the HSAM sample with and without a coating on the sound

**TABLE 2.** Density of Perforated Samples of HSAM with a Ceramic Coating

Order No.	Diameter of opening in the sample, mm	Perforation area, %	Sample density, kg/m <sup>3</sup>
1	1.5	5	160
2	2.0	10	150
3	2.5	12.5	140
4	4.0	15	130



**Fig. 3.** Sound absorption coefficient of HSAM with a ceramic coating: 1) sample without perforation with coating No. 16 (one layer); 2) perforated sample with coating No. 16 (one layer); 3) same (three layers); 4) perforated sample with coating No. 11 (one layer).

absorption coefficient  $\alpha$  were studied using a high-level interferometer (HLI) at an affiliate of the Federal State Unitary Enterprise Central Aerohydrodynamic Institute.

It was found that as the perforation area of the HSAM samples with coating No. 16 increases to 15% the sound-absorption coefficient increases to 0.9 – 1 in the frequency range from 1 to 4.5 kHz. The sound-absorption coefficient of the HSAM samples with coating No. 16 lies in the range 0.7 – 0.8 without perforations and 0.7 – 0.8 with perforations. The sound absorption coefficient of HSAM samples with coating No. 11 lies in the range 0.7 – 0.8 without perforations and 0.8 – 0.9 with perforations (Fig. 3).

The sound absorption coefficient of HSAM samples with three layers of coating No. 16 decreases from 0.8 to 0.5 – 0.6. As coating thickness increases, sound absorption decreases because the porosity of the sound absorbing material decreases. It was found that the structure of the HSAM, coating thickness and perforation of coated HSAM samples all strongly affect the sound absorption coefficient, which is also affected by the coating composition but to a lesser degree.

The results obtained for the technological properties, temperature stability and phase stability (to 750°C,  $\tau$ -500) of HSAM with a coating were analyzed. The composition of the ceramic coating was optimized on the basis of the data obtained. The composition No. 16 based on ceramic-forming polymer PKSZ-21 with filler consisting of fine silicon carbide powder is optimal. It was found that the sound absorption coefficient of HSAM with coating No. 16 lies in the interval 0.7 – 0.8;  $\alpha = 0.8 - 1$  for the same samples with perforation (perforation area 15%).

The acoustic, mechanical and thermal tests established that the ceramic coating No. 16 could find application for developing sound-absorbing structures consisting of multilayer perforated panels of heat-and-sound absorbing materials.

## REFERENCES

1. V. Dmitriev and A. Munin, "Ecological problems of civilian aviation," *Nauka i Proizvodstvo*, No. 2, 15 – 17 (2003).

2. V. M. Kuznetsov and A. G. Munin, "Problems of fighting airplane noise," in: *80th Anniversary of the Central Aerohydrodynamic Institute, Anniversary Journal* [in Russian], pp. 14 – 18.
3. A. F. Sobolev, N. M. Solov'ev, and R. D. Filippova, "Expansion of the sound-absorption frequency band of linings of the propulsion systems of jet aircraft," *Akustich. Zh.*, **4**(1), 146 – 152 (1995).
4. S. S. Solntsev, V. T. Minakov, V. A. Rozenenkova, et al., "Complex temperature-stable protective coating for ceramo-ceramic composite materials," in: *Proc. 18th Conference on Temperature-Stable Functional Coatings* [in Russian], TGPU im. L. N. Tolstogo, Tula (2001), Part 2, pp. 8 – 10.
5. S. S. Solntsev, *Protective Technological Coatings and Refractory Enamels* [in Russian], Mashinostroenie, Moscow (1984), pp. 220 – 241.
6. V. T. Minakov and S. S. Solntsev, "Ceramo-ceramic composites — 21th century materials," in: *Aviation Materials: Selected Works 1932 – 2002, Anniversary Scientific-Technical Symposium* [in Russian], MISIS, VIAM, Moscow (2002), pp. 122 – 130.